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Evaluation of Refractory/Austenitic Bimetal Combinations

Second Quarterly Progress Report

By

R. W. Buckman, Jr. and J. L. Godshall

Prepared for

National Aeronautics and Space Administration

Lewis Research Center

Space Power Systems Division

Under Contract NAS 3-7634

Astronuclear Laboratory

Westinghouse Electric Corporation

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Second Quarterly Progress Report

Covering the Period

September 22, 1965 to December 22, 1965

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-7634

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FOREWORD

This report was prepared by personnel of the Astronuclear Laboratory of the Westinghouse Electric Corporation under Contract NAS 3-7634. This work is administered under the direction of the Nuclear Power Technology Branch of the National Aeronautics and Space Administration with Mr. P. L. Stone acting as Technical Manager.

This work is being administered at the Astronuclear Laboratory by R. T. Begley with J. L. Godshall and R. W. Buckman, Jr. serving as principal investigators. This report covers the work performed during the period September 22, 1965 to December 22, 1965. Other Westinghouse Astronuclear Laboratory personnel contributing to this work include Messrs. D. R. Stoner and E. Vandergrift.

EVALUATION OF REFRACTORY/AUSTENITIC BIMETAL COMBINATIONS

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ABSTRACT

Evaluation of the bond integrity of as-explosive bonded refractory/austenitic bimetal composites continued using tensile tests, hardness traverses, cold rolling, and metallographic analysis. Nine combinations were selected for complete evaluation of thermal stability. Assembly of ultra-high diffusion annealing systems for the isothermal exposures was completed and the diffusion annealing and thermal exposure runs were initiated.

Author

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I. INTRODUCTION

This is the second quarterly progress report under Contract NAS 3-7634, "Evaluation of Refractory/Austenitic Bimetal Combinations". The objective of this program is to evaluate the compatibility of various refractory/austenitic bimetal combinations after isothermal and cyclic thermal exposure. The refractory/austenitic bimetal composites were fabricated by explosive bonding.

During this quarter, evaluation of the bond integrity of the as-explosive bonded bimetal composites continued. Tensile tests, hardness traverses, cold rolling, and metallographic analysis were utilized in conjunction with non-destructive ultrasonic and dye penetrant testing to determine the propensity of the interfacial bond to delaminate under various mechanical stresses. Nine austenitic/refractory bimetal composites were selected with the approval of the cognizant NASA project officer for detailed evaluation.

Assembly of ultra-high vacuum diffusion annealing systems for the isothermal exposures was completed and the 1600 hour runs at 1400, 1500, and 1600°F were initiated. Thermal cycling and creep rupture testing equipment have been installed and are being calibrated and adjusted for optimum performance.

II. PROGRAM STATUS

A. STARTING MATERIAL

1. Selection of Composites for Evaluation — The statement of work for this study specified that nine explosively bonded austenitic/refractory bimetal composites are to be evaluated according to the program outlined in the First Quarterly Report.[1] During preparation of the composites, seven additional combinations were produced, raising to sixteen the total number of bimetal combinations. These combinations are listed in Table 1. The nine combinations underlined were selected, with the approval of the cognizant NASA project manager, for detailed evaluation. However, all sixteen combinations were subjected to

TABLE 1 - As-Received Explosively Bonded Refractory/Austenitic Bimetal Compositions

<u>Cb/321</u>*
<u>Cb/Inconel 600</u>*
<u>Cb-1Zr/347</u>*
<u>Cb/347</u>*
<u>Ta/Inconel 600</u>*
<u>Ta/347</u>*
<u>Ta/321</u>*
FS-85/347
<u>FS-85/321</u>*
FS-85/Inconel 600
<u>Cb-1Zr/321</u>*
T-222/Inconel 600
T-222/321
Ta/Hastelloy N
T-222/347
FS-85/Hastelloy N

*Selected for full evaluation.

ultrasonic and dye penetrant testing, metallographic examination, and reverse bend testing. All sixteen combinations will be given the isothermal exposures at 1400, 1500, and 1600°F, for 1600 and 2700 hours.

Selection of the nine combinations was predicated primarily on metallurgical considerations, and the integrity of the explosively bonded interface was of secondary importance since all combinations could be satisfactorily explosively bonded. The tendency for brittle-intermetallic compound formation between the refractory metal and the components of the austenitic material at relatively low temperatures (1700-1800°F) restricts the exposure temperature for the bimetal couples. The primary use of the austenitic/refractory bimetals will be as tubing which will be cold reduced to final size from an explosively bonded bimetal tube blank. Processing tubing will require frequent intermediate annealing treatments because of the high rate of work hardening of the austenitic materials. The low work hardening rates of the pure Cb and Ta, and the Cb-1Zr permit large reductions without intermediate annealing. The temperature for stress relief annealing of the austenitics is sufficiently low that interdiffusion between the base alloys should be insignificant. However, to anneal the high rate of work hardening, high strength columbium and tantalum base alloys, FS-85 and T-222, would require heating to temperatures in the range of 2000 to 2200°F for FS-85 and 2400 to 2600°F for T-222. The latter range, of course, approaches the melting point of the austenitic materials. Even the former range is unacceptable since excessive interdiffusion would undoubtedly occur and the brittle intermetallic zone formed would be likely to fracture during subsequent processing. The use of lower annealing temperature and longer annealing times would prove to be impractical. For these reasons, virtually all bimetal combinations incorporating the above alloys were eliminated from the detailed evaluation. The FS-85/321 combination was selected to be evaluated in detail as the ninth combination in order to include one high strength refractory metal austenitic bimetal combination in the study.

2. Metallography — Samples taken from the center and either end of each 6 inch x 12 inch x 0.09 inch composite sheet were examined metallographically in an attempt to establish the end at which explosive detonation occurred. This information was not recorded when the composites were formed. The microstructural examination revealed a wide variation

in the geometry of the interface. Examples of the typical interface geometries are shown in Figure 1 and can be described as:

a. Little or no wave form, tending toward planar interface (Figure 1a).
b. Combination planar and wave form, with variation in amplitude of wave (Figure 1b).
c. Crested wave peak exhibiting a "hooked" effect, and uniform amplitude (Figure 1c).
d. Wave form of varying frequency (Figure 1d).

The combinations a, b, and c exhibited excellent bonding characteristics,and no delamination of the as-received material was detected with ultrasonic inspection. The FS-85/321 combination (Figure 1d) exhibited large unbonded areas of the as-received material during ultrasonic (U. T.) inspection. However, there does not appear to be any correlation between interface geometry and bonding integrity. Of course, it must be emphasized that all the metallographic samples were taken from areas of the sheet exhibiting sound bonds. Samples cut from areas which showed U. T. indications separated when sectioned and exhibited a planar interface. Generally, there was excellent agreement between metallographic and ultrasonic test results with respect to identification of unbonded areas.

3. Hardness — Tukon hardness traverses were taken on all sixteen composites at 1, 2, 3, 5, 10, and 20 mils from the interface in each of the two metals. Diamond pyramid hardness of each of the base metals was taken midway between the edge and interface. These data are recorded in Table 2 and the hardness traverse data are plotted in Figures 2, 3, 4, and 5. All of the austenitic metals generally showed significant work hardening near the interface and the hardness level of the austenitic metal has also been significantly increased. The as-annealed hardness of the austenitic material was approximately 130-145 DPH. The hardness of the austenitic material after explosive bonding is 234-297 DPH, a significant increase. Dieter[2] has discussed in some detail the shock hardening of metals which occurs without appreciable changes in dimensions. The high hardness at the interface is attributed to localized flow of these surfaces during explosive bonding. Data are not available on the

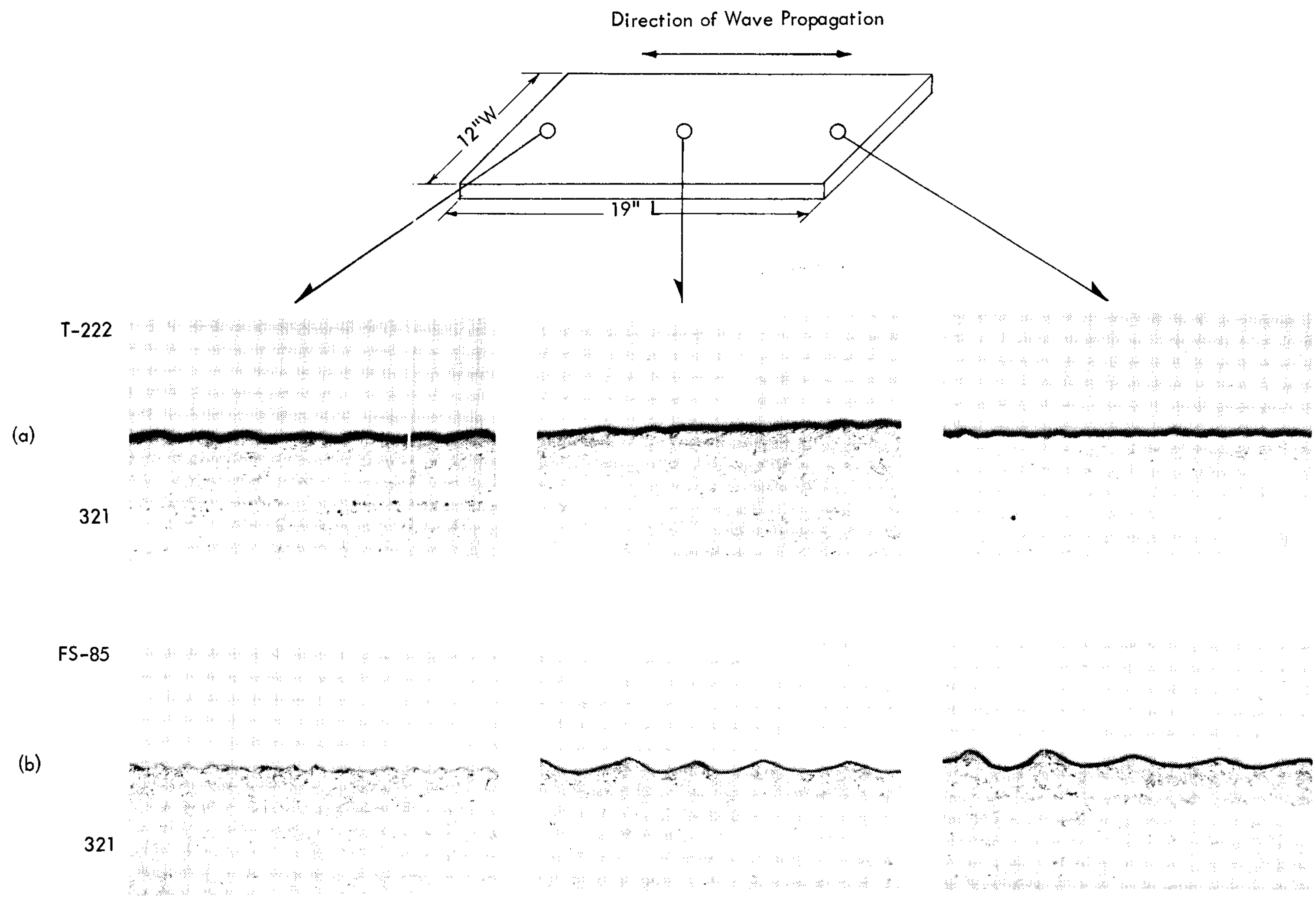


FIGURE 1 - Photomicrographs of Refractory/Austenitic Bimetal Composites Showing Geometry of Interface. (Note: Shadows at Interface Caused by Relief Between Austenitic and Refractory Metal Component are not Separations) Mag. 100X (Reduced 30% in Reproduction)

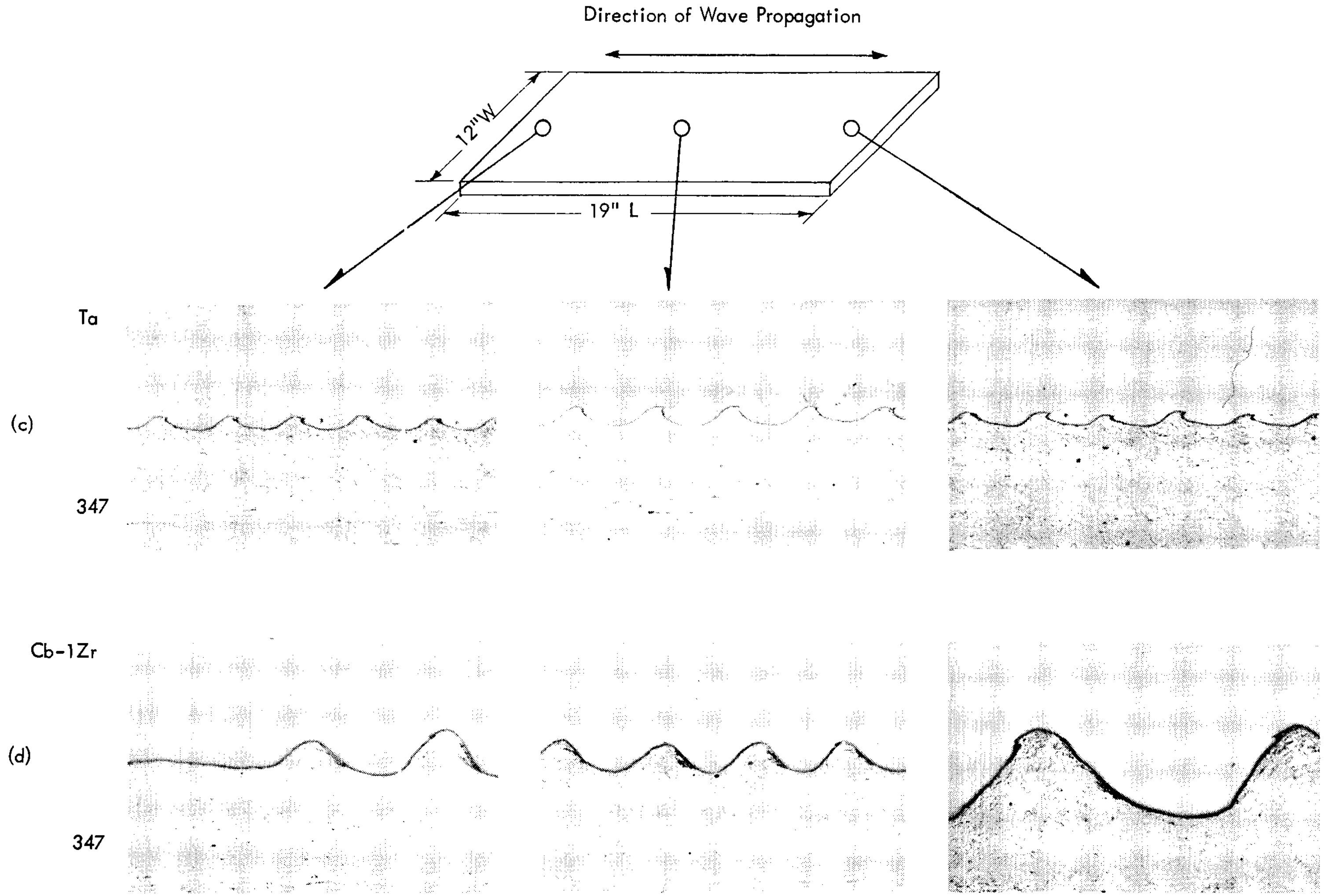


FIGURE 1 (continued) - Photomicrographs of Refractory/Austenitic Bimetal Composites Showing Geometry of Interface. (Note: Shadows at Interface Caused by Relief Between Austenitic and Refractory Metal Components are not Separations) Mag. 100X (Reduced 30% in Reproduction)

TABLE 2 - Hardness of As-Bonded Refractory/Austenitic Bimetal Composites

Composition	Knoop Hardness Distance from Interface (mils) 1	2	3	5	10	20	Base Metal Hardness DPH[a]
Cb	118	120	121	127	119	107	97
347	434	458	430	394	336	315	277
Cb	138	133	128	116	125	108	82
321	421	398	381	409	305	293	263
Cb	126	129	119	112	118	112	105
Inconel 600	374	300	295	313	295	236	226
Cb-1Zr	168	174	171	167	171	150	120
347	463	409	409	425	364	313	247
Cb-1Zr	154	164	153	148	152	142	118
321	398	390	381	364	295	292	243
FS-85[b]	261	298	295	263	282	231	232
347	332	326	318	241	278	219	231
FS-85	298	310	289	269	336	278	251
Inconel 600	413	409	451	355	358	267	258
FS-85	265	318	285	282	303	267	245
321	430	405	390	390	303	318	269
FS-85	326	308	271	305	293	298	232
347	413	390	329	329	271	271	234
FS-85	298	303	292	293	278	254	226
321	488	477	405	409	425	342	297
FS-85	323	313	293	280	271	241	224
Hastelloy N	451	390	390	390	326	287	227

TABLE 2 - Hardness of As-Bonded Refractory/Austenitic Bimetal Composites (Continued)

Composition	Knoop Hardness						Base Metal Hardness DPH(a)
	Distance from Interface (mils)						
	1	2	3	5	10	20	
Ta	201	207	207	207	189	171	172
321	473	473	443	405	368	326	259
Ta	216	193	191	180	183	188	172
Inconel 600	378	351	358	351	318	247	240
Ta	186	186	160	178	176	183	146
Hastelloy N	584	654	552	413	361	329	264
T-222	358	398	358	374	313	295	290
Inconel 600	430	409	374	342	321	278	228
T-222	390	381	385	351	351	310	291
321	468	447	387	409	324	274	234
T-222	378	385	413	394	355	335	282
347	488	417	447	458	355	303	285

(a) Base Hardness Level, Measurement Made Equidistant Between Interface and Surface

(b) Traverse Taken at Area of "No-Bond"

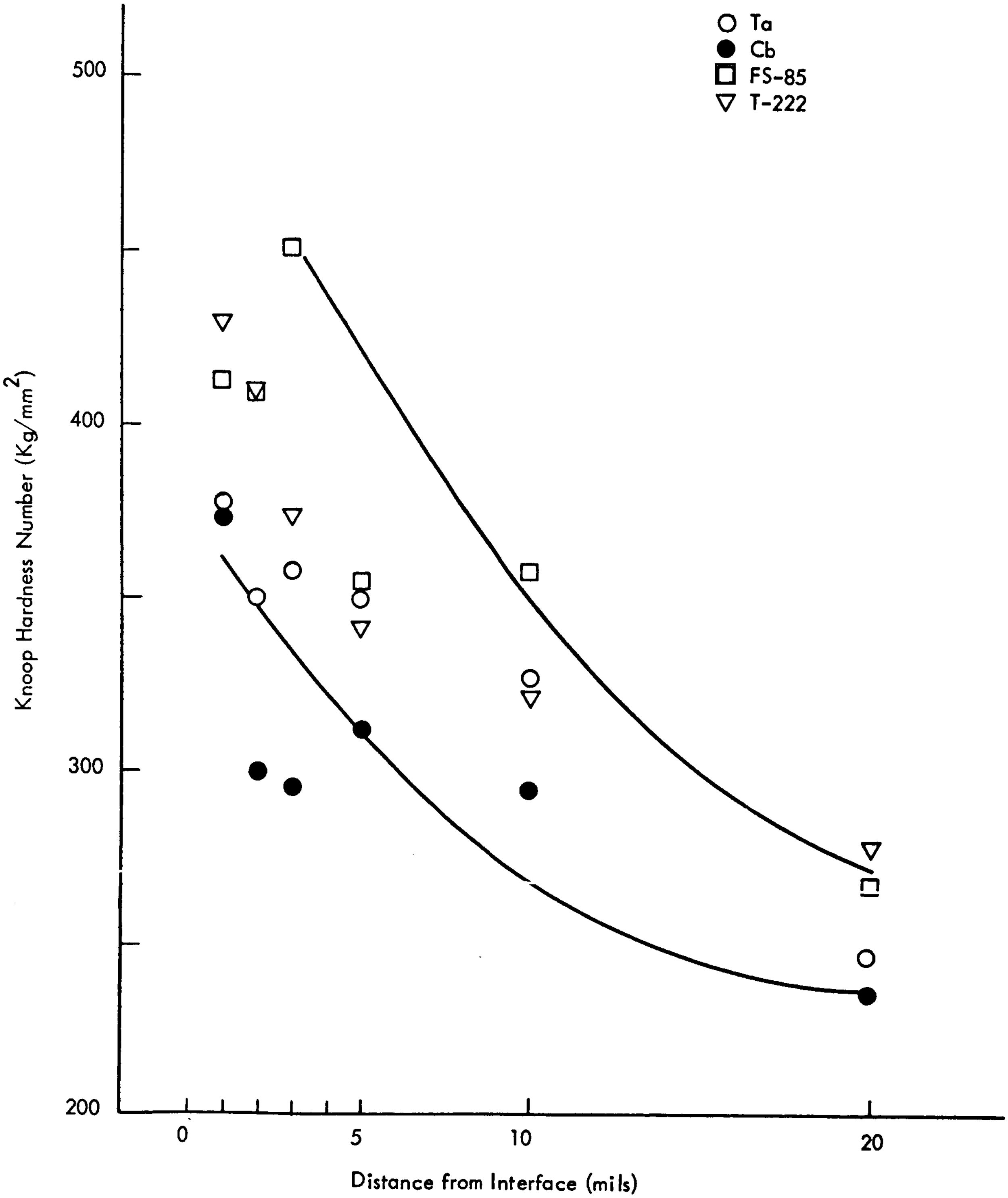


FIGURE 2 - Hardness Traverses for Inconel 600 Explosively Bonded to the Various Refractory Metal Alloys.

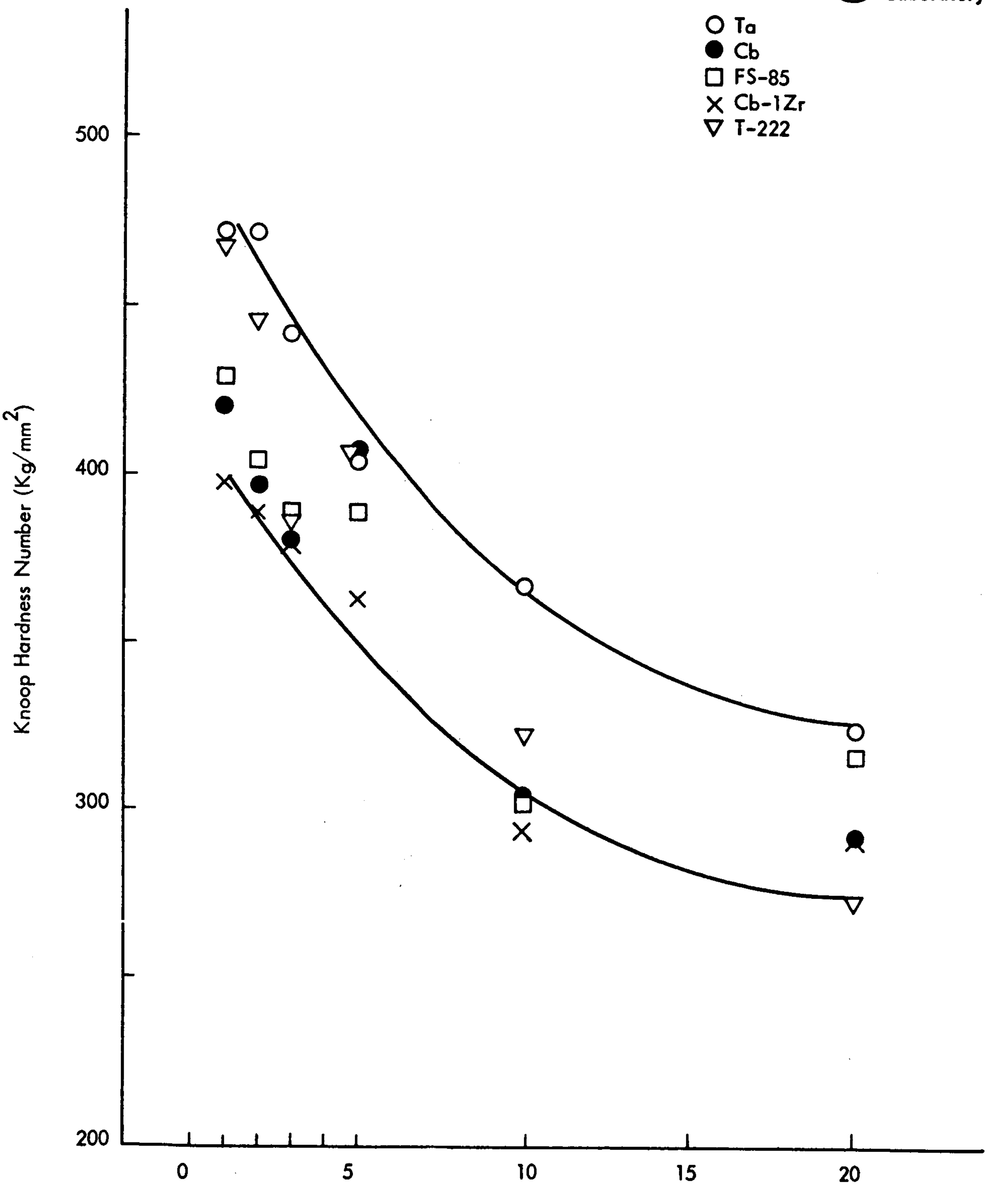


FIGURE 3 - Hardness Traverses for Type 321 Stainless Steel Explosively Bonded to the Various Refractory Metal Alloys.

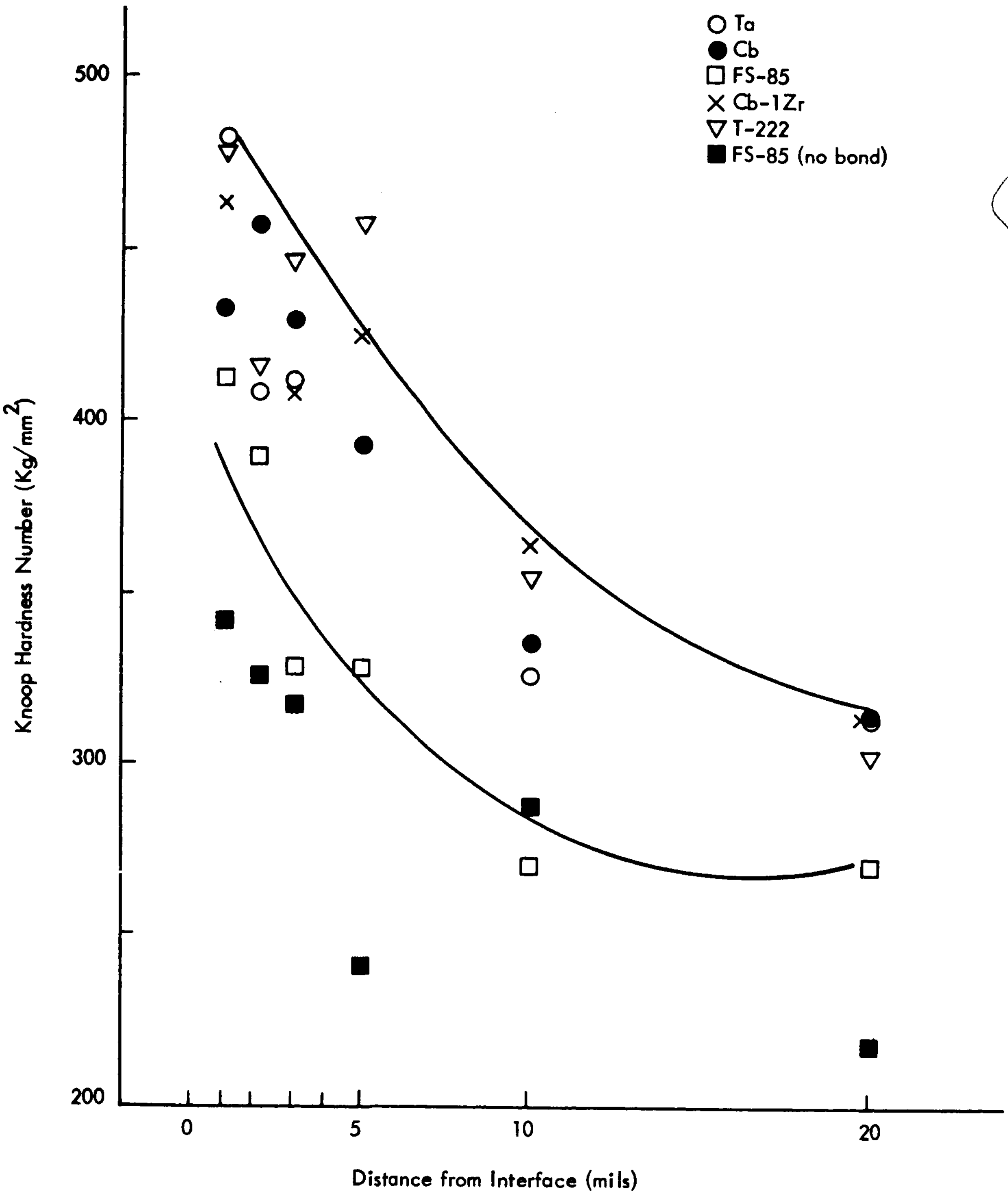


FIGURE 4 - Hardness Traverses for Type 347 Stainless Steel Explosively Bonded to the Various Refractory Metal Alloys.

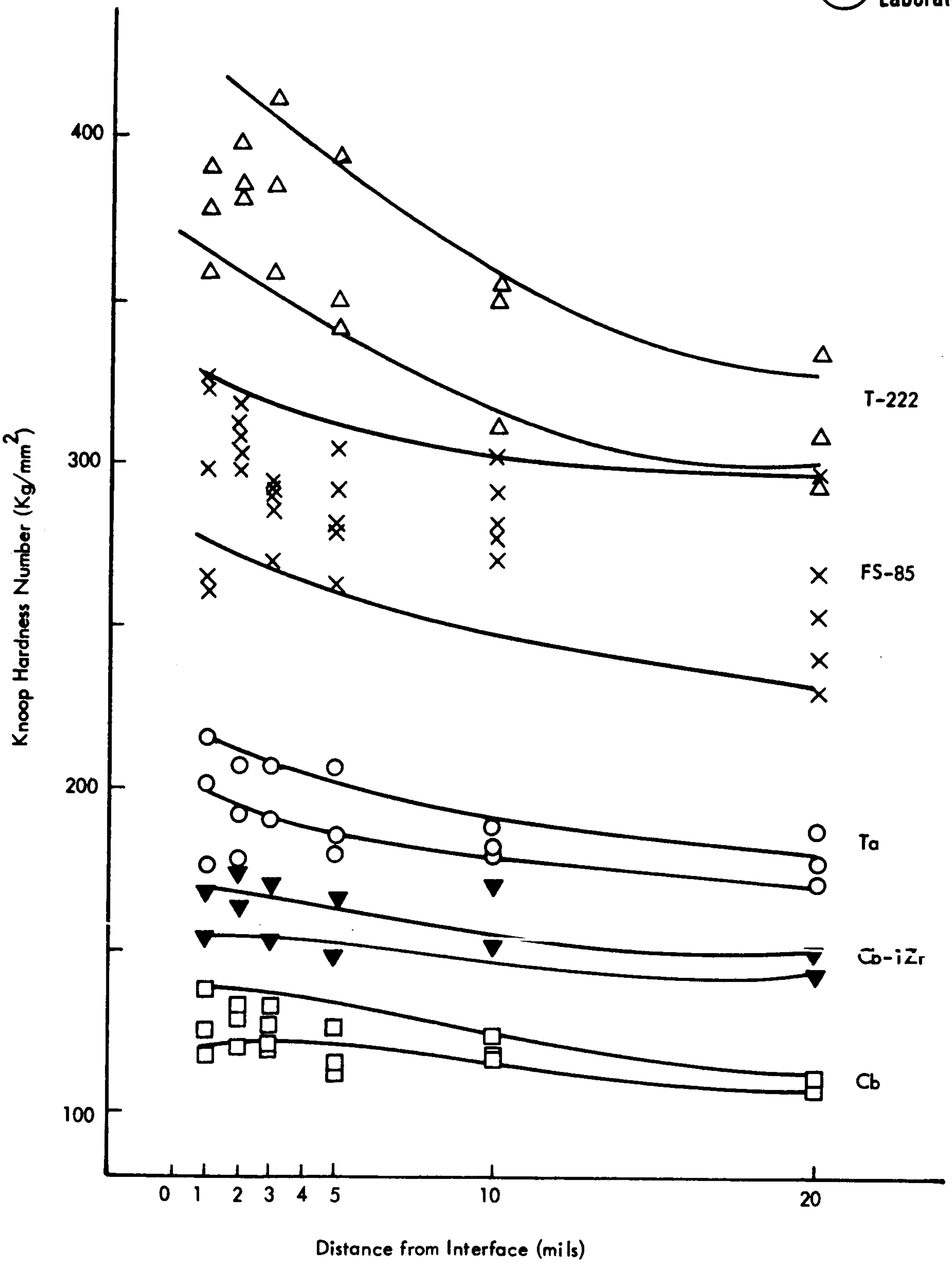


FIGURE 5 - Hardness Traverses for the Refractory Metal Alloys Explosively Bonded to the Austenitic Materials.

mechanical properties of the refractory metal sheet prior to bonding. The low work hardening rate of Cb, Ta, and Cb-1Zr was reflected by the observed small increase in hardening near the interface, and the bulk hardness is representative of that for material of the specified chemistry and condition of heat treatment. The hardness of T-222 and FS-85 in the fully recrystallized condition should be approximately 280 and 186 DPH respectively. The bulk hardness of the FS-85 and T-222 shows a hardness increase, due to the high pressure shock during bonding,and a hardness gradient near the locally deformed interface. The data scatter for the FS-85 is greater than for the other refractory metals indicating the possibility of loss of control of the bonding process. This observation is of particular significance in view of the fact that the FS-85 exhibited generally the poorest bonding characteristics.[1]

4. <u>Tensile Tests</u> — Ten of the refractory/austenitic bimetal combinations were tensile tested on a Wiedemann, Mark G, 60,000 pound capacity universal testing machine. Duplicate samples were tested at room temperature using a constant cross-head motion of 0.05 in/in/minute. The specimens were lightly etched to delineate the refractory/austenitic interface and the behavior of each specimen was recorded photographically through the yield point, ultimate strength, and fracture. After the test, each sample was polished to accurately locate the interface and the reduction in area of the refractory and austenitic components was determined. The results of the tensile tests are shown in Table 3 and the reduction in area for each metal is shown in Table 4. Examination of 32 frame/second photographic film showed that none of the samples delaminated prior to fracture. Figure 6 shows the Cb/Inconel 600 composite at the time of fracture and one film frame after fracture.

In Table 5 are listed the room temperature mechanical properties for the austenitic materials prior to explosive bonding. Similar data for the refractory metals were not available but will be determined. As discussed earlier, there has been an increase in hardness of the austenitic material as a result of the explosive bonding. This hardness increase corresponds to an increase in yield strength of from $(65-91)\times10^3$ psi. This work hardening induced primarily into the austenitic material is probably the explanation for the similarity in strength level

TABLE 3 - Tensile Test Data on Refractory/Austenitic Bimetal Composites*

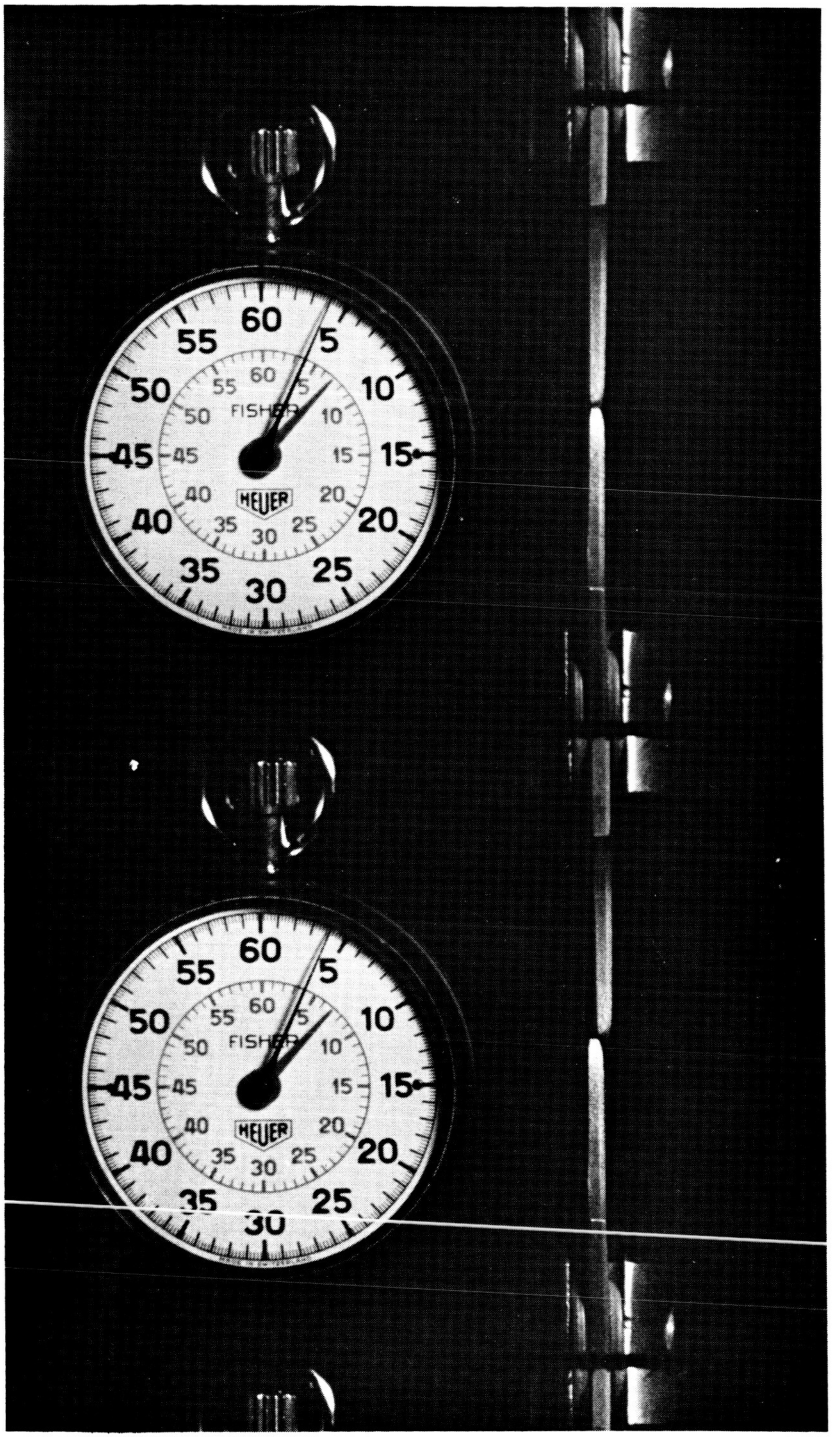
Composition	0.2% Yield Strength (ksi)	Ultimate Strength (ksi)	Elongation (%)	Red. In Area (%)
Cb/321	74.9 74.1	82.0 80.6	41.0 43.0	66.0 61.3
Cb/Inconel 600	75.1 73.2	81.0 80.9	24.0 30.0	67.8 69.3
Cb-1Zr/321	82.8 81.8	90.9 90.7	22.0 22.0	58.9 58.8
Cb/347	78.3 80.2	85.8 88.0	35.0 32.0	54.1 60.0
Ta/Inconel 600	90.3 89.3	94.0 93.7	21.0 21.0	68.2 58.4
Ta/347	86.9 85.1	94.4 92.5	27.0 31.0	59.3 56.2
Ta/321	92.0 90.1	96.6 94.8	20.0 31.0	56.5 58.6
FS-85/321	98.6 97.5	102.1 101.4	26.0 28.0	56.7 51.9
Cb-1Zr/347	86.2 85.7	92.1 92.1	24.0 29.0	62.3 50.8
T-222/321	87.2 88.8	99.5 98.2	38.0 34.0	54.8 55.0

* Room Temperature Tests

TABLE 4 - Tensile Test Reduction in Area - Refractory, Austenitic, and Total

Composition	Reduction in Area (%) Refractory	Austenitic	Total
Cb/321	77.3	56.2	66.0
	56.1	59.3	61.3
Cb/Inconel 600	80.9	59.2	67.8
	81.1	56.1	69.3
Cb-1Zr/321	61.2	55.0	58.9
	60.3	56.3	58.8
Cb/347	50.1	54.8	54.1
	66.6	65.3	60.0
Ta/Inconel 600	63.8	68.7	68.2
	66.7	52.8	58.4
Ta/347	67.4	56.8	59.3
	48.5	60.1	56.2
Ta/321	62.1	56.8	56.5
	59.5	55.5	58.6
FS-85/321	47.7	56.9	56.7
	40.0	56.2	51.9
Cb-1Zr/347	50.9	65.5	62.3
	41.5	52.5	50.8
T-222/321	56.9	47.2	54.8
	49.3	55.6	55.0

(a) At Fracture



(b) 1 Frame After Fracture

FIGURE 6 - Failure of Cb/Inconel 600 Composite During Tensile Testing (Photographed at 32 Frames Per Second).

TABLE 5 - Mechanical Properties[a] of Austenitic Materials Used for Fabrication of Composites.

Composition	0.2% Y.S. (psi)	UTS (psi)	% Elongation	Hardness R_B	Hardness DPH
AISI 316	44,000	82,400	49	81	155
AISI 321	33,600	84,200	51	78	149
AISI 347	41,900	86,200	46	80	153
Inco 600	31,100	87,900	45	71	130

(a) Vendor furnished certified test results.

for all the composites, which do not appear to be significantly affected by the composition of the refractory metal component.

5. Cold Rolling — The resistance of the bond to severe mechanical deformation was examined by cold rolling each of the sixteen composites. Samples were rolled with the direction of rolling both longitudinal and transverse to the direction of wave propagation during bonding. All of the samples were ultrasonically and dye penetrant checked to insure that there were no defects in the material prior to reduction. The 0.090 inch thick composites were reduced to 0.015-0.018 inch thick by multiple rolling passes. FS-85/321 failed in the transverse rolling direction. All of the remaining sheets reduced with no visual defects. The as-rolled sheets were examined by simple hand flexing. The FS-85/321 sheet exhibited audible clicks, while the other integrally bonded combinations did not.

B. DIFFUSION ANNEALING

Interdiffusion between the austenitic and refractory metal is to be determined after exposures of 1600 and 2700 hours at 1400, 1500, and 1600°F. The long time elevated temperature exposures are being done under ultra high vacuum (1 x 10^{-8} torr) conditions in all metal sealed, bakeable, sputter ion pumped systems. During the current report period, the 1600 hour runs at 1400, 1500, and 1600°F were initiated. The time accumulation on each run at the end of this current quarterly period is:

1400°F - 1140 hours (Scheduled completion 1/10/66)
1600°F - 900 hours (Scheduled completion 2/5/66)
1500°F - 24 hours (Scheduled completion 2/27/66)

Each specimen (1/4 inch x 3/4 inch x 0.09 inch thick) was belt sanded, etched in a 15% HNO_3-3%HF-H_2O solution, dye penetrant checked for delamination, re-cleaned, re-etched, then wrapped in tantalum foil prior to insertion into the vacuum system. A Pt/Pt-13%Rh thermocouple is embedded in each load and is used to monitor temperature.

In addition to the diffusion annealing samples, tensile, creep rupture, and 1 inch x 8 inch strips of each of the nine bimetal combinations (See Table 1) selected for detailed study are being exposed for 1600 hours at 1500°F to determine the effect of this exposure on mechanical properties. Duplicates of these test specimens will be exposed on a second run at a later date since the size of the vycor tube, in which the specimens are contained, limited the number of samples which could be exposed at one time. Also included in this first 1500°F exposure run are samples of 1/2 inch diameter bimetal (316-Cb) tubing produced from a co-extruded tube blank and from an explosively bonded tube blank.

The power supply for the furnace which contained the thermal exposure load malfunctioned after approximately 72 hours of testing which resulted in a temperature overshoot of 75°F, thus terminating this test prematurely. Replacement specimens will be made, the furnace reloaded, and the test re-run.

C. THERMAL CYCLING

The apparatus[1] for conducting the thermal cycling test (600-1350°F) was assembled during this report period and checkout of the system was started. A photograph of the system is shown in Figure 7. The oxygen gage, which will be used to monitor the quality of the helium gas is the only piece of instrumentation that remains to be added to the system.

Bimetal test strips, nominally 1 inch x 8 inch x 0.090 inch thick will be heated from 600-1350°F in five minutes, held at temperature for fifteen minutes, and cooled to 600°F in approximately thirty seconds. This cycle will be repeated a total of twenty times for each complete test. Three strips are positioned symmetrically in an induction coil and are prevented from bending by means of a molybdenum restraint fixture. The specimens are heated by radiation from a stainless steel susceptor coupled to a 450 kc R.F. generator. Helium gas is directed at the refractory metal side to accelerate cooling. Heating is done in vacuum (<1 x 10^{-5} torr). During this period, the vacuum system was checked out, the temperature uniformity was established on the test specimens, and preliminary runs were made to determine if the

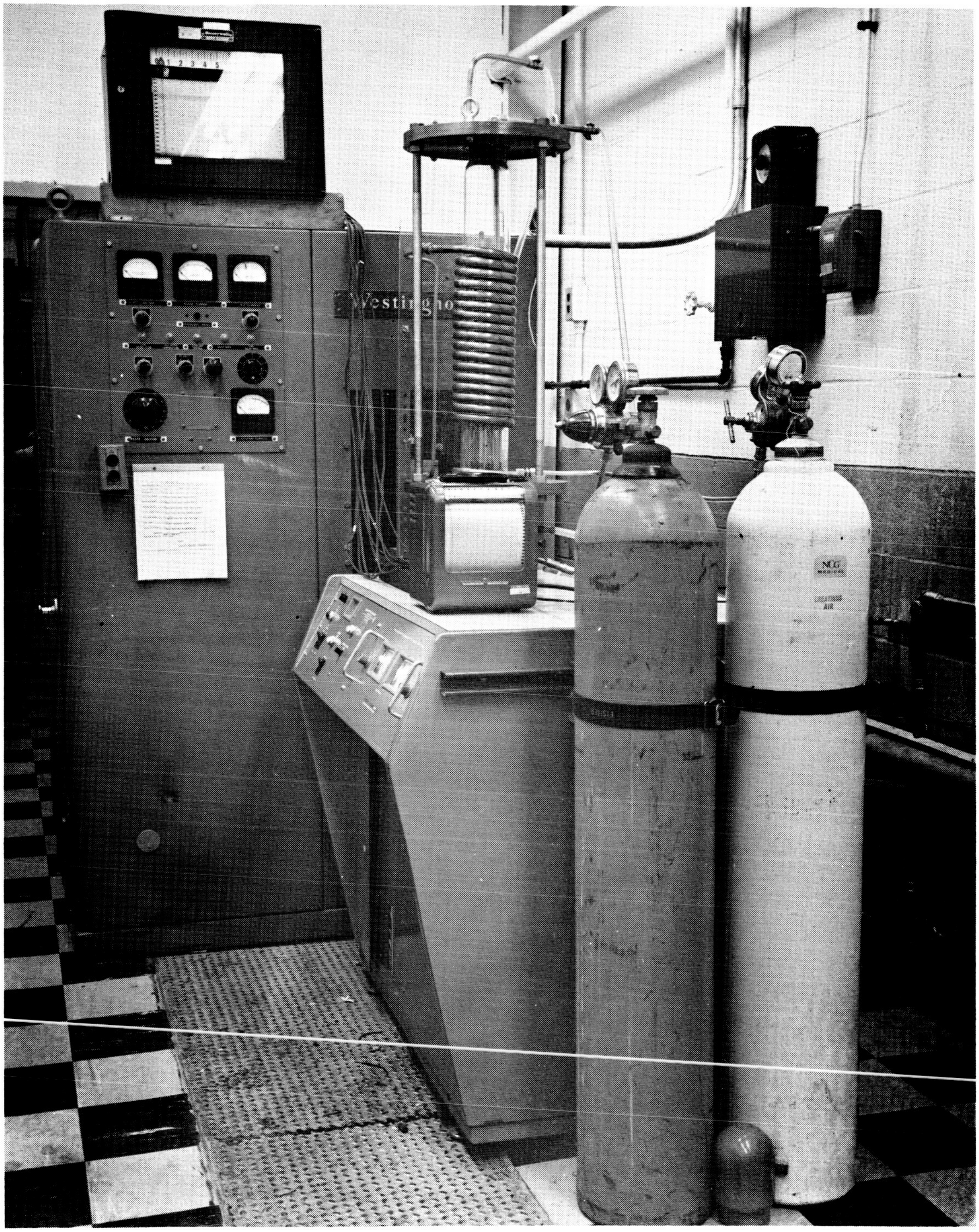


FIGURE 7 - Thermal Cycling Apparatus

pre-selected heating and cooling rate could be obtained with the experimental set-up. All temperature measurements were made at the austenitic-refractory metal interface using 0.005 inch Pt/Pt-13%Rh thermocouples. The symmetry of heating was very good with less than 10°F temperature difference from specimen to specimen. The temperature distribution from top to bottom was 49°F which is within the ±25°F specified in the work statement. However, the axial temperature distribution will be improved by changing the position of the test specimen holder within the susceptor. Cooling from 1350°F to 600°F was accomplished in ninety seconds, and consumed approximately 16 ft.^{3} of helium. This amount of helium useage would require a bottle of helium for each 20 cycles. It appears that the cooling rate from 1350°F to 600°F within thirty seconds will be achievable by reducing the mass of the restraint fixture and increasing the diameter of the jets of the perforated gas delivery tube from 0.04 inch diameter to 0.06 inch thereby increasing the helium flow rate to the test strips. The main vacuum valve on the system developed a leak and therefore a shutdown was necessary. A replacement valve has been ordered.

D. CREEP-RUPTURE TESTING

Both of the ultra-high vacuum creep-rupture units were received and installed. The units were qualified by the vendor, i.e., at a system pressure of 1×10^{-8} torr with the furnace at 3500°F. The units after installation were re-qualified and again, with the furnace at 3500°F as measured by an optical pyrometer, the system pressure was less than 1×10^{-8} torr. The pressure on each system was on the 10^{-8} torr range prior to energizing the ion pump, which attests to the leak tightness of the system which had not been pumped on for approximately two weeks. Base pressure at room temperature was 2×10^{-10} torr. (Note: System had been baked by the vendor and had not been vented to atmosphere prior to qualification test at WANL.) Pressure measurements were made with a hot filament nude ionization gage.

The associated components for the creep-rupture test systems which include the loading system, grips, and tungsten weight have been received. The Honeywell three mode precision controllers which were scheduled for shipment on December 3, 1965, have been re-scheduled for shipment February 18, 1966. Test specimen temperature uniformity and initial system checkout will continue but initiation of creep-rupture testing will be delayed until the controllers are received.

III. FUTURE WORK

During the next period, the following will be accomplished:

1. Stress-rupture testing will be initiated.
2. Preliminary checkout of thermal cycling apparatus will be completed and testing will be initiated on as-received materials.
3. The 1600 hour diffusion annealing runs will be completed and the 2700 hour runs initiated.
4. Metallographic examination of as-received and thermally exposed bimetal composites will continue.

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2. G. E. Dieter, "Hardening Effect Produced with Shock Waves", 1960 Seminar Strengthening Mechanisms in Solids, ASM.

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